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(54) Diaphragm aluminum alloy plates and their preparation

(57) Aluminum alloy plates best suited for diaphragm molding of composite thermoplastic resin material are provided. The alloy composition consists essentially of, in % by weight, 2.0-6.0% of Mg, 0.0001-0.1% of Be, 0.001-0.15% of Ti alone or in combination with 0.0001-0.05% by weight of B for grain refinement, 0-0.2% of Fe, 0-0.2% of Si, 0-0.05% of Mn, 0-0.05% of Cr, 0-0.05% of Zr, and 0-0.05% of V as impurities, and the balance of aluminum. Intermetallic compound particles and recrystallized grains have a specific size. The plate manufacturing process involves the steps of DC casting, heating, hot rolling, optional intermediate annealing, cold rolling, and optional final annealing, or the steps of continuous casting, optional intermediate annealing, cold rolling, and optional final annealing.

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TITLE OF THE INVENTION

Diaphragm Molding Aluminum Alloy Plates and Their Preparation

This invention relates to an aluminum alloy for use in diaphragm molding of composite thermoplastic resin material.

BACKGROUND OF THE INVENTION

The demand for FRP (fiber-reinforced plastics) having high strength and high modulus is increasing especially in the aerospace field. Composite materials having carbon or similar fibers impregnated with epoxy thermosetting resins were first developed, but these FRP structures were low in heat resistance and impact resistance.

A new type of fiber-reinforced composite material in which fibers were impregnated with thermoplastic resins instead of the thermosetting resins were recently developed and found an increasing application as aerospace related composite structures. Commonly used thermoplastic resins are polyether ether ketones (PEEK) and polyarylene ketones (PAK). These FRP structures cannot be molded such as by pressing at room temperature since thermoplastic resins remain hard at room temperature. Therefore, in order to mold FRP structures, they must be softened by heating prior to molding, and after molding, hardened again by cooling down to room temperature. More particularly, fibers impregnated with thermoplastic resins are interposed between plates of a material adapted for hot forming such as aluminum and then subjected to hot pressing or diaphragm molding (compressed air molding). The diaphragm molding often uses aluminum plates.

Research efforts are concentrated on superplastic aluminum alloys exhibiting an elongation of 300% or more at temperatures of 400°C or higher. Known superplastic aluminum alloys include Al-78% Zn, Al-33% Cu, Al-6% Cu-0.4% Zr (Supral), Al-Zn-Mg-Cu alloys (7475 and 7075), and Al-2.5-6% Mg-0.05-0.6% Zr alloys. It is empirically known that these superplastic aluminum alloys except Supral show a low elongation at low temperatures and require heating to a temperature of the order of 0.8Tm (Tm: melting point in °K) to provide sufficient elongation. This heating temperature is extremely higher than the molding temperature of PEEK and similar thermoplastic resins.

In an attempt to use superplastic aluminum alloys in diaphragm molding of thermoplastic resin base composite materials, the inventors found the following problems. Superplastic aluminum alloys themselves are generally adapted for molding at high temperatures in excess of 450°C, but less amenable to molding in the temperature range of from 200 to 450°C associated with the diaphragm molding of thermoplastic resin base composite materials. Further, they are slow in strain rate under optimum conditions (elongation rate under a temperature condition optimized for maximum elongation) and thus require an increased molding time, resulting in inefficient commercial manufacture.

The only aluminum alloy which has been commercially successful in diaphragm molding of thermoplastic resin base composite materials is Al-6% Cu-0.4% Zr (Supral). Supral is not only less efficient in production and costly due to complexity of its production process, but also difficult to recycle due to the high concentration of Cu although the diaphragm molding process uses disposable aluminum alloy plates.

SUMMARY OF THE INVENTION

The present invention has been made in order to overcome the above-mentioned problems, and its object is to

provide an aluminum alloy plate which is amenable to hot diaphragm molding of thermoplastic resin base composite materials. Another object is to provide an aluminum alloy plate which shows improved properties, especially elongation for diaphragm molding of thermoplastic resin base composite materials in the temperature range of from 200 to 450°C. A further object is to provide a simple process for preparing the aluminum alloy plate.

Making investigations on an aluminum alloy containing Mg as a main additive component, the inventors have found that the aluminum alloy can be tailored optimum for the diaphragm molding of thermoplastic resin base composite materials by controlling the content of alloy components, especially the content of Mg and impurity elements, the size of intermetallic compound particles after final annealing, and the shape of grains which are recrystallized prior to or during diaphragm molding.

According to the present invention, there is provided an aluminum alloy having a composition consisting essentially of, in % by weight, 2.0 to 6.0% of Mg, 0.0001 to 0.01% of Be, 0.001 to 0.15% of Ti alone or in combination with 0.0001 to 0.05% by weight of B for grain refinement, up to 0.2% of Fe, up to 0.2% of Si, up to 0.05% of Mn, up to 0.05% of Cr, up to 0.05% of Zr, and up to 0.05% of V as impurities, and the balance of aluminum. Preferably, the alloy composition further contains 0.05 to 2.0% of Cu or 0.2 to 2.5% of Zn or both.

The alloy most often takes the form of rolled sheets which are best suited for use in diaphragm molding of composite thermoplastic resin material. Intermetallic compounds of aluminum with impurities have a maximum particle size of 10 μm. Recrystallized grains, if any, have a ratio L/T of up to 1.5 provided that in a cross section parallel to a plate rolling direction, the grains have a mean grain size L in the rolling direction and a mean grain size T in a plate thickness direction. The recrystallized

grains encompass both the grains which are recrystallized during final annealing and the grains which are recrystallized during diaphragm molding. These two situations will be understood from a common practice of diaphragm molding wherein the molding plates are preheated prior to actual molding. That is, the diaphragm molding method includes a preheating step and a molding step. It safficies that recrystallization has taken place in the molding plates prior to the start of the molding step.

The aluminum alloy plate is prepared by semi-continuously casting an alloy having the above-defined composition; heating the alloy at 450 to 580°C for 1/2 to 48 hours; hot rolling the alloy at an initial temperature of 400 to 530°C; and cold rolling the alloy to a draft of at least 15% prior to final recrystallization. Intermediate annealing may be carried out between the hot rolling step and the cold rolling step or midway the cold rolling step (that is, between cold rolling steps).

Alternatively, the plate is prepared by continuously casting an alloy having the above-defined composition, and cold rolling the cast alloy to a draft of at least 15% prior to final recrystallization. Intermediate annealing may be carried out before or midway the cold rolling step.

In either process, the cold rolled plate may be finally annealed if desired.

DETAILED DESCRIPTION OF THE INVENTION

The aluminum alloy of the present invention has an alloy composition consisting essentially of, in % by weight,

2.0 to 6.0% of Mg,

0.0001 to 0.01% of Be,

0.001 to 0.15% of Ti alone or in combination with 0.0001 to 0.05% by weight of B for grain refinement,

up to 0.2% of Fe, up to 0.2% of Si, up to 0.05% of Mn, up to 0.05% of Cr, up to 0.05% of Zr, and up to 0.05% of V as impurities, and

the balance of aluminum and incidental impurities.

The reason of limiting the content of alloying components is first described.

Mg: 2.0 to 6.0%

Magnesium is effective in improving the warm working of the alloy since it promotes work softening and dynamic recrystallization. Less than 2.0% of Mg is insufficient to impart strength and warm workability whereas alloys containing more than 6.0% of Mg are difficult to produce due to poor hot and cold rolling performance. The Mg content is thus limited to 2.0 to 6.0%.

Be: 0.0001 to 0.01%

Beryllium is effective in preventing oxidation of Mg upon melting as well as mold galling during diaphragm molding. Less than 0.0001% of Be is ineffective whereas no further benefit is obtained beyond 0.01% of Be.

Ti: 0.001 to 0.15%, B: 0.0001 to 0.05%

Titanium is added alone or along with boron for the purpose of cast ingot grain refinement. Less than 0.001% of Ti is ineffective whereas proeutectic $TiAl_3$ particles will crystallize out in excess of 0.15% of Ti. Similarly, less than 0.0001% of B is ineffective whereas TiB_2 particles will crystallize out in excess of 0.05% of B.

Cu & Zn:

Copper and zinc are effective in improving strength, increasing stacking fault energy, and reinforcing the dislocation cell structure during working. Less than 0.05% of Cu or less than 0.2% of Zn is ineffective whereas more than 2.0% of Cr or more than 2.5% of Zn causes a lowering of corrosion resistance and will precipitate along the grain boundary, resulting in a lowering of warm elongation.

Impurities, Fe, Si, Mn, Cr, Zr, V, etc.:

Fe, Si, Mn, Cr, Zr, V, and other impurities, if present in substantial contents, will form coarse intermetallic compounds such as Al-Mn, Al-Mn-Fe, Al-Si, and Al-Fe-Si compounds during casting. Once formed, such intermetallic

compounds can never be removed by subsequent working and heat treatments. If the intermetallic compounds have a particle size of more than 10 µm, they will become a starting site of fracture during diaphragm molding, which is detrimental for diaphragm molding. Therefore, Fe should be 0.2% or less, Si 0.2% or less, Mn 0.05% or less, Cr 0.05% or less, Zr 0.05% or less, and V 0.05% or less.

Other incidental impurities should be 0.1% or less in total.

The metal structure is limited for the following reason.

Intermetallic compound:

When the aluminum alloy plate is used for diaphragm molding, intermetallic compounds based on impurities should have a maximum particle size of 10 µm. Intermetallic compound particles having a particle size of more than 10 µm will become a fracture starting site during diaphragm molding, adversely affecting diaphragm molding.

Recrystallized grains:

The aluminum alloy can have recrystallized grains if final annealing is effected to induce recrystallization at the last stage of aluminum alloy plate manufacturing process. Alternatively, if aluminum alloy plates have not been recrystallized at the last stage, recrystallization would be induced by the heat applied during diaphragm molding.

In the temperature range of from 200 to 450°C during diaphragm molding of thermoplastic resin base composite materials, deformation occurs through "transgranular deformation" and "cyclic dynamic and static recrystallization". If recrystallized grains have a flat shape during diaphragm molding, the "transgranular deformation" would be accompanied by local stress concentration leading to fracture, and "dynamic and static recrystallization" would cause uneven recrystallization,

also inviting local stress concentration and hence, fracture.

A ratio L/T is used as a measure representative of the flatness of recrystallized grains during diaphragm molding, provided that recrystallized grains have a mean grain size L in a plate rolling direction and a mean grain size T in a plate thickness direction in a cross section parallel to a plate rolling direction and perpendicular to the plate surface. According to the present invention, the recrystallized grains should have a ratio L/T of from 1 to 1.5.

The aluminum alloy plates of the present invention is prepared by the following method.

Casting:

Semi-continuous or direct chill (DC) casting is commonly used.

Ingot heating:

Cast ingots are heated at 450 to 580°C for about 1/2 to about 48 hours. Heating may be conducted in a single stage or in multiple stages in combination with homogenization. In the case of multiple stage heating, it suffices that the maximum temperature among the stages meet the above-mentioned condition. Heating below the range is ineffective for homogenizing purposes and makes it difficult to use the hot rolling starting temperature of 400°C. Heating beyond the range would form coarser intermetallic compounds to adversely affect diaphragm molding and sometimes cause eutectic melting.

Hot rolling:

Heated ingots are then hot rolled into plates at an initial temperature of 400 to 530°C. Temperatures of lower than 400°C are too low for hot rolling purposes. Temperatures of higher than 530°C tend to cause edge cracks due to the elevated temperature embrittlement of Mg.

Intermediate annealing:

If desired, hot rolled plates may be subject to intermediate annealing. It may be either batchwise annealing at 250 to 450°C for 1/2 to 24 hours or continuous annealing at 300 to 580°C without holding or for 5 minutes or less. The intermediate annealing may be carried out between the hot rolling and cold rolling steps or midway the cold rolling step.

Cold rolling:

Hot rolled plates are then cold rolled at a draft of 15% or more. With a cold rolling draft of less than 15%, recrystallization would not occur or would occur unevenly upon the final recrystallizing step. This is undesirable for diaphragm molding. The upper limit of draft is usually 95% in view of rolling cost. The draft by cold rolling should reach 15% or more prior to final recrystallization.

Where intermediate annealing is carried out midway the cold rolling step, that is, between cold rolling steps, it is essential that the final cold rolling step (after the intermediate annealing) achieves a draft of 15% or more.

Final recrystallization:

In general, cold rolled plates are finally annealed for recrystallization.

Diaphragm molding is carried out near the softening temperature of thermoplastic resins, that is, from 200 to 450°C. An assembly of aluminum plates having a thermoplastic resin layer sandwiched therebetween is placed in a molding machine at an elevated temperature where the resin is held until it is heated to the predetermined temperature, during which recrystallization occurs in the aluminum plates. Alternatively, a preheater is used to preheat the assembly. If the preheating temperature is above 250°C so that recrystallization may occur during preheating, then final annealing to induce a recrystallized structure may be omitted from the plate manufacturing process.

The temperature and holding time of annealing, if employed, are not limited insofar as recrystallization can occur. Either continuous or batchwise annealing may be employed. In general, batchwise annealing is at 250 to 400°C for 1/2 hour or longer, and continuous annealing is at 350 to 550°C without holding or for at most 180 seconds.

The process has been described as starting from semi-continuous casting (DC casting) although continuous casting (CC) may also be used. Continuous casting eliminates the need for plate heating and hot rolling.

Briefly stated, the aluminum alloy plates of the present invention are produced by the following two processes.

Process 1	Process 2
semi-continuous casting	continuous casting
ingot heating	(CC plate heating)
hot rolling	cold rolling
(intermediate annealing)	(intermediate annealing)
cold rolling	cold rolling
(intermediate annealing)	(final annealing)
cold rolling	
(final annealing)	

* The steps in parentheses are optional.

EXAMPLE

Examples of the present invention are given below by way of illustration and not by way of limitation.

Alloys 1 to 8 having the composition shown in Table 1 were DC cast into ingots having a cross section of 1000 mm x 400 mm. The ingots were homogenized at 530°C for 10 hours, heated at 500°C for 3 hours, and then hot rolled at a starting temperature of 450°C into plates of 4 mm thick. After hot rolling, the plates were cold rolled into plates

of 1 mm thick (cold rolling draft: 75%) and finally annealed at 350°C for 2 hours.

Separately, alloys having essentially the same composition as Alloys 1 and 3 were CC cast into plates of 3.0 mm thick x 400 mm wide. The plates were cold rolled into plates of 1 mm thick (cold rolling draft: 66.6%), and finally annealed at 350°C for 2 hours. These plates are designated Alloys 1′ and 3′.

Table 1 Alloying elements (wt%)

Alloy	Mg	Ti	B	Be	Mn	Cr	Zr	V	Cu	Zn	Fe	Si	casting	Remarks
1	5.7	0.01	0.0015	0.0008	-	-	-	-	-	-	0.03	0.04	DC	Invention
1'	5.6	0.02	0.0019	0.0007	-	-	-	-	-	-	0.04	0.05	CC	Invention
2	4.6	0.01	0.0010	0.0010	0.02	0.01	-	-	0.61	-	0.09	0.05	DC	Invention
3	4.4	0.02	0.0004	0.0012	-	-	-	-	0.30	1.75	0.06	0.03	DC	Invention
3'	4.4	0.02	0.0001	0.0008	-	-	-	-	0.33	1.81	0.04	0.04	CC	Invention
4	2.8	0.01	0.0003	0.0009	-	0.01	-	-	-	0.83	0.10	0.09	DC	Invention
5	3.7	0.02	0.0005	0.0013	0.34	-	-	-	0.02	-	0.28	0.21	DC	Comparison
6	4.5	0.01	0.0005	-	-	0.15	0.05	0.06	-	-	0.46	0.25	DC	Comparison
7	3.0	0.02	0.0150	0.0008	0.10	0.19	-	-	0.35	0.82	0.19	0.15	DC	Comparison
8 *	4.4	0.03	-	-	0.69	0.18	-	-	0.02	0.04	0.28	0.15	DC	Comparison

* JIS 5083

The finally annealed plates were sectioned in a longitudinal direction (parallel to a rolling direction) and perpendicular to the major surface (in a thickness direction). The sections were polished and observed by means of an image analysis apparatus to measure the maximum size of intermetallic compound particles. The results are shown in Table 2.

Table 2

Alloy		Maximum size of intermetallic compound after final annealing, µm
1	Invention	2.0
1'	Invention	1.8
2	Invention	6.3
3	Invention	4.8
3'	Invention	3.1
4	Invention	7.6
5	Comparison	21.8
6	Comparison	18.5
7	Comparison	16.6
8	Comparison	23.9

The intermetallic compound particles in the alloys of the invention are smaller in size than those in the comparative alloys.

Next, the aluminum alloy plates were examined for their amenity to diaphragm molding of thermoplastic resin base composite materials. An elongation was measured by a warm tensile test at 400°C. The plates were held at 400°C for 10 minutes before the start of the tensile test. The gage length was 10 mm. The results are shown in Table 3.

Table 3

Alloy		Elongation in hot (400°C) tensile test at strain rate (/sec.) 10^{-3}	10^{-2}	10^{-1}
1	Invention	384	340	288
1'	Invention	392	340	291
2	Invention	357	332	271
3	Invention	359	324	265
3'	Invention	358	326	269
4	Invention	322	296	234
5	Comparison	251	214	166
6	Comparison	202	161	115
7	Comparison	208	178	139
8	Comparison	215	173	131

The alloys of the invention show a higher elongation at the elevated temperature than the comparative alloys. The difference becomes increased with an increase in strain rate.

A stack of eight 0.1-mm layers of PEEK resin-impregnated carbon fibers was sandwiched between a pair of cold rolled plates of 1 mm thick. Some runs used the plates which had been finally annealed at 350°C for 2 hours. The remaining runs used the plates which had not been finally annealed, but could undergo recrystallization by heating during bulging. Each assembly was held at 400°C for 5 minutes and then subjected to diaphragm molding by effecting bulging to a diameter of 100 mm, for determining the bulging height. After holding at the elevated temperature for 5 minutes in the bulging machine (before bulging), the grain size was measured to determine a ratio L/T, that is, (mean grain size in a rolling direction)/(mean grain size in a thickness direction). The results are shown in Table 4.

Table 4

Alloy	Final anneal	Grains (μm) L	T	L/T	Bulging under 4 atm. Time/height	8 atm. Time/height
1	Yes	39	31	1.26	29/62	3/60
	No	35	26	1.35	31/61	4/60
1'	Yes	32	25	1.28	29/63	4/60
2	Yes	58	47	1.23	35/59	5/56
3	Yes	65	56	1.16	33/58	4/56
3'	Yes	58	50	1.16	32/60	4/57
4	Yes	68	50	1.36	31/56	3/55
5	Yes	45	25	1.80	38/47	4/44
6	Yes	61	26	2.35	39/33	5/28
7	Yes	54	22	2.45	38/38	6/36
8	Yes	48	21	2.29	44/46	6/45
	No	50	23	2.17	53/46	6/42

* Time/height = min./mm.

To examine superplasticity, the room temperature strength of the aluminum alloy plates after warm working was compared with typical prior art Al-Mg alloy structural materials. From the plates subjected to 50% warm tension at 400°C, JIS No. 5 specimens were cut and subjected to a tensile test at room temperature. The results are shown in Table 5. It is to be noted that the prior art alloys designated Nos. 9, 10 and 11 had not been subjected to 50% warm tension at 400°C

Table 5

Alloy	Mg (wt%)	Tensile strength (kg/mm^2)	Yield point (kg/mm^2)	Remarks
1	5.7	33.3	16.8	Invention
2	4.6	30.9	15.3	Invention
3	4.4	29.5	14.5	Invention
4	2.8	23.0	10.6	Invention
9	2.6	19.8	9.4	Prior art 5052-0
10	4.5	29.9	14.8	Prior art 5182-0
11	4.5	30.5	16.2	Prior art 5083-0

For the same content of Mg, the alloys of the invention maintained at least equal strength even after warm working as compared with the prior art alloys which had not been warm worked. Therefore, the alloys of the invention are useful superplastic forming materials.

There have been described aluminum alloy plates adapted for use in diaphragm molding of thermoplastic resin base composite materials. The plates contribute to efficient production of thermoplastic resin base composite structures.

The aluminum alloy plates of the present invention are not only suitable for diaphragm molding, but may themselves be subjected to superplastic forming and warm working into a variety of complexly configured articles including electric control housings, meter housings, chassis of VCR and other electric appliances, automobile bodies, gasoline tanks, and oil pans.

Although some preferred embodiments have been described, many modifications and variations may be made thereto in the light of the above teachings. It is therefore to be understood that within the scope of the appended claims, the invention may be practiced otherwise than as specifically described.

CLAIMS:

1. An aluminum alloy plate for use in diaphragm molding of composite thermoplastic resin material, having an alloy composition consisting essentially of, in % by weight,

2.0 to 6.0% of Mg,

0.0001 to 0.01% of Be,

0.001 to 0.15% of Ti alone or in combination with 0.0001 to 0.05% by weight of B for grain refinement,

up to 0.2% of Fe, up to 0.2% of Si, up to 0.05% of Mn, up to 0.05% of Cr, up to 0.05% of Zr, and up to 0.05% of V as impurities, and

the balance of aluminum and incidental impurities,

wherein intermetallic compounds of impurities have a maximum particle size of 10 μm, and grains which are recrystallized prior to or during diaphragm molding have a ratio L/T of up to 1.5 provided that the grains have a mean grain size L in a plate rolling direction and a mean grain size T in a plate thickness direction.

2. The aluminum alloy plate of claim 1 wherein the alloy composition further contains at least one member of 0.05 to 2.0% of Cu and 0.2 to 2.5% of Zn.

3. A process for preparing an aluminum alloy plate for use in diaphragm molding of composite thermoplastic resin material, comprising the steps of:

semi-continuously casting an alloy having the composition as defined in claim 1 or 2,

heating the alloy at 450 to 580°C for 1/2 to 48 hours,

hot rolling the alloy at an initial temperature of 400 to 530°C, and

cold rolling the alloy to a draft of at least 15% prior to final recrystallization.

4. The process of claim 3 which further comprises the step of intermediate annealing between the hot rolling step and the cold rolling step.

5. The process of claim 3 or 4 which further comprises the step of intermediate annealing midway the cold rolling step wherein the last cold rolling achieves a draft of at least 15%.

6. A process for preparing an aluminum alloy plate for use in diaphragm molding of composite thermoplastic resin material, comprising the steps of:

continuously casting an alloy having the composition as defined in claim 1 or 2, and

cold rolling the cast alloy to a draft of at least 15% prior to final recrystallization.

7. The process of claim 6 which further comprises the step of intermediate annealing before the cold rolling step.

8. The process of claim 6 which further comprises the step of intermediate annealing midway the cold rolling step wherein the last cold rolling achieves a draft of at least 15%.

9. A diaphragm molding plate formed of an aluminum alloy consisting essentially of, in % by weight,

2.0 to 6.0% of Mg,

0.0001 to 0.01% of Be,

0.001 to 0.15% of Ti alone or in combination with 0.0001 to 0.05% by weight of B for grain refinement,

up to 0.2% of Fe, up to 0.2% of Si, up to 0.05% of Mn, up to 0.05% of Cr, up to 0.05% of Zr, and up to 0.05% of V as impurities, and

the balance of aluminum and incidental impurities,

wherein intermetallic compounds of impurities have a maximum particle size of 10 μm, and grains which are recrystallized prior to or during diaphragm molding have a ratio L/T of up to 1.5 provided that the grains have a mean grain size L in a plate rolling direction and a mean grain size T in a plate thickness direction.

10. The plate of claim 9 wherein the alloy composition further contains at least one member of 0.05 to 2.0% of Cu and 0.2 to 2.5% of Zn.

11. An aluminium alloy plate, substantially as hereinbefore described in accordance with any one of Alloys 1, 1', 2, 3, 3' and 4 of the Example.

12. A process for preparing an aluminium alloy plate, substantially as hereinbefore described in accordance with any one of Alloys 1, 1', 2, 3, 3' and 4 of the Example.

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